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Olive waste oil an economical source for biodiesel

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ABSTRACT

The increasing energy demands along with the expected depletion of fossil fuels have promoted search for alternative fuels that can be obtained from renewable energy resources. The paper found that olive waste oil (pomace olive oil) one of the cheapest biodiesel feedstock collected from mills in Egypt used to produce biodiesel. In this olive waste oil extracted from olive waste samples, using hexane as an extraction solvent. The oil converted to biodiesel (methyl ester) in two step process: acid-catalyzed esterification followed by alkali catalyzed transesterification. It found that the oil content in olive waste-represents approximately 15.20% by mass. The results of the two step transesterification process shows yield 95.50% obtained at 50% v/v of methanol/oil ratio, 60ºC temperature with 1.00% by weight KOH catalyst.

Key words: Olive waste oil, pomace olive oil, waste to energy, biodiesel, transesterification.

1. INTRODUCTION

The search for alternative energy resources to supplement or replace fossil fuels has increased in the recent years because of the increased environmental concern, energy security, and fast depletion of fossil fuel resources. Biodiesel has become more attractive as it is nontoxic, environmental friendly, and made from renewable resource (Meher *et al.,* 2006 and Vyas *et al.,* 2010). The most commonly used vegetable oils for biodiesel production are soybean, sunflower, palm, rapeseed and canola oils. The major drawback with biodiesel is the high cost of feedstock which leads to the high price of biodiesel (Issariyakul *et al.,* 2008). Hence, the use of edible oils for biodiesel production is not feasible. One possible way to reduce the production cost of biodiesel is to use nonedible oils (Mohamed *et al.,* 2012 and Koberg and Gedanken 2012), waste frying oil (Math *et al.,* 2010 and Leung *et al.,* 2006) and poultry fats (Reddy *et al.,* 2006) as raw materials. Also, utilizing biomass residues for biodiesel production will reduces the contribution of biofuels production to food price. Biomass energy defined as the biological material such as agricultural residues, animal wastes, forestry and wood processing residues. Biomass is seen as few renewable indigenous, widely dispersed energy sources and one of the most promising, renewable energy sources. It is, among the renewable forms of energy, the major source of the primary energy supply (Mckendry, 2002). Pressing olive fruits for oil extraction produces annually a large amount olive cake, the solid by-product of olive fruit after pressing for extraction of oil. It comprises a significant fraction, 13-30% of the olive fruit (Hamdan *et al.,* 1993 and Kiritsakis and Markakis 1988). The olive cake contains a small oil fraction (10 to 15%). This amount of oil is extracted and consumed only in soap industry. Therefore, utilization of this source for energy production through a systematic method of raw material management could be significant in terms of energy cost savings and in terms of pollution control. The price of olive waste oil is 2-3 times cheaper than virgin vegetable oil (Tanawannapong *et al.,* 2013). The usage of olive waste oil as a reactant for biodiesel synthesis not only helps the disposal problem but also reduces the production cost of biodiesel.

The most common way to produce biodiesel is by transesterification which refers to a catalyzed chemical reaction involving vegetable oil and an alcohol to yield fatty acid methyl esters (biodiesel) and glycerol. Triglycerides, as the main component of vegetable oil consist of three long chain fatty acids esterifies to a glycerol structure. When triglycerides react with an alcohol (e.g., methanol, the three fatty acid chains are with the methanol to yield fatty acid methyl esters. Glycerol is produced as a by-product for biodiesel production, chemically catalyzed processes, including alkali catalyzed and acid catalyzed ones have proved to be more practical than the energy catalyzed process. An alkali catalyzed process can achieve high purity and yield of biodiesel product in a short time (Corro *et al.,* 2011). However, it is very sensitive to the purity of the reactants. On the other hand, acid catalysis is more efficient when the amount of free fatty acids in the oil exceeds 1% (Liu, 1994 and Zhang *et al.,* 2003). However, the reaction requires a longer reaction time (About 4 the times needed by alkali catalyzed reaction (Lotero *et al.,* 2005), and run at higher reaction temperature. Moreover, the water formation during the acid catalyzed reaction of the free fatty acids with the methanol can stop the esterification reaction of free fatty acids before it could be completed. Also, biodiesel contaminated with water can cause engine corrosion or reaction with glycerides to produce soaps and glycerol. Additionally, acid catalysts are difficult to recycle and operate at high temperatures and give rise to serious environmental and corrosion problems (Canakci and Gerpen 2005). The objective of this work was to apply two step transesterification approaches for converting olive wastes oil collected from mills in Egypt to fatty acid methyl ester (Biodiesel).

2. MATERIAL AND METHODS

Materials: olive wastes were obtained from an oil factory located in Horticultural Research Institute, Agricultural Research Center, Giza, Egypt. All chemicals used in the current study were obtained from Sigma Chemical Company (England, London Ltd., Pools).

Oil extraction: Olive waste was not suitable for processing because of its high moisture content. Olive wastes were taken and dried in an oven for 1h at 105ºC, then ground in a Sonic Japan Blender (Model: SB 2525) solvent extraction at boiling point of 40-60ºC. Upon the completion of extraction, the extracts to a round bottom flask attach an evaporation apparatus for solvent removal. The extracted oil was weighed and the percentage oil content was calculated using Equation:

$$\text{Oil content (\%)} = \text{weight of extracted oil (g)} \,/\, \text{weight of sample (g)} \times 100 \qquad (1)$$

The oil extractions were carried out within 30 min of grinding, since the free fatty acid content of the extracted oil was to be determined. The extraction procedure was repeated until sufficient oil for transesterification as well as feed stock analysis was obtained (A.O.A.C. 2000).

Biodiesel Production: Olive waste represents a valuable low cost biomass source for biodiesel manufacturing. Such low price raw material is of special interest it will significantly reduce the production costs of biodiesel. In this way, the price of biodiesel will be in with petroleum based diesel (Zhang *et al.,* 2003). For Biodiesel production two step processes, acid-catalyzed esterification followed by alkali catalyzed transesterification used KOH concentration and methanol ratio on the biodiesel yield investigated.

First step: Acid catalyzed esterification: the pretreatment of olive waste oil was carried out in 250ml three necked round bottom reactor equipped with magnetic stirrer, a reflux condenser, thermometer and stopper to remove samples. The reactor was placed in water bath, the water bath sets on heating and mixing plate. A known amount of olive waste oil was poured into the reaction flask and heated at 60ºC. The methanol was added to the heated at (40v %) and stirred for a few minutes. Sulfuric acid was then added to the mixture. Two different H_2SO_4 were used: 1ml and 5ml. the mixture was agitated under reflux for 4hr.After the reaction completed, the mixture was allowed to settle in a 500ml separation funnel for 3h. The bottom layer which contains methanol-water H_2SO_4 fractions was removed. The upper layer consist of olive waste oil having lower content of free fatty acid and impurities were purified by washing with hot sodium bicarbonate solution (5% w/v), to neutralize excess H_2SO_4 and reduce the acidity, and allowed to settle for 3h for complete phase separation. Again the bottom layer was drain. The acidity of the bottom layer was detected with phenolphthalein indicator. The procedure was repeated until the drained layer becomes basic. The treated oil was then washed with distilled water and the mixture was allowed to still 3hr for complete separation between the two layers. The procedure was repeated until the washing water had a pH value that was similar to that of distilled treated oil was then dried by gentle heating for complete water removal. Finally, the acid value of the oils product was determined.

Second step: Alkali-catalyzed transesterification: The transesterification reaction was carried out by using the same experiment setup of pretreatment step. The reactor was initially charged with treated oil (oil with low free fatty acid), then heated to the required temperature. The potassium hydroxide pellets was dissolve in methanol and the mixture was heated oil the flask. Stirring velocity was kept at 800rpm. The reaction was timed as soon as the potassium hydroxide- methanol solution was fed to the reactor. The reaction was kept at a desired temperature for 4hr. After the reaction the mixture was poured into separating funnel and allowed to settle for 3hr. The ester layer was separated by gravity and located in the upper layer. The glycerol, extra methanol and undesired products in the lower layer were decanted. The biodiesel layer was washed with distilled water for complete removal of excess methanol and any catalyst traces. The washing step was repeated until the washing was neutral. The ester layer was then dried by gentle heating to obtain the refined biodiesel. The degree of oil conversion and the biodiesel yield were recorded. The transesterification reaction was temperature at 60ºC for 4 hr, methanol ratio 30 v % and KOH concentration (1.00 wt% of olive waste oil).

Acid value determination: The acid value of the initial olive waste oil or after the free fatty acid esterification reaction was determined according to A.O.A.C (2000).

Peroxide value determination: The peroxide value of the initial olive waste oil was determined according to A.O.A.C. (2000).

Determination of the % conversion of free fatty acids: The conversion of the free fatty acid is defined as the fraction of free fatty acid that reacted during the esterification with methanol. The conversion free fatty acid (% CFFA) was determined from the acid value ratio using the following Equation:

$$\% \text{ CFFA} = Ai\text{-}Af/Ai \times 100 \qquad (2)$$

Where Ai is the initial acid value of the olive waste oil; Af is the final acid value of the olive waste oil after the free fatty acid esterification reaction.

Chemical analysis of the biodiesel produced: the fatty acid methyl esters content in the biodiesel obtained was measured by GC analysis according to the EN 14103 test method, using a GC-2014 Shimadzu Chromatograph provided with a FID detector. The biodiesel sample was added to the standard solution containing methyl heptadeconoate. The mixed sample was analyzed by GC. The fatty acids methyl ester (FAME) content was determined using the following Equation:

$$\text{FAME (\%)} = \Sigma A\text{-}As/As \times CsVs/M \times 100 \qquad (3)$$

Where ΣA is the sum of peak areas of methyl ester ($C_{14:0}$-$C_{24:1}$); as is the peak area of methyl heptadeconoate; Cs is the concentration of methyl heptadecanoate; Vs is the volume of standard solution; M is the amount of biodiesel sample.

Biodiesel density and kinematic viscosity determinations: Density of the samples was calculated form the following Equation:

P= Pref x gs (4)

Where P is the density; P ref is the reference density; gs is the specific gravity.

The kinematic viscosity of the produced biodiesel was measured acceding to the Suguy *et al.,* (1996) using Brookfield DVIII model digital rheometer at 40ºC by measuring the time required for a volume of liquid to flow and under gravity through a Cannon-Fensake Style glass Capillary tube.

Flash and heating number: Flash and heating number of the samples were measured in the temperature range of 60 to 190ºC by an automated Pensky- Martens closed cup apparatus (ASTM D941)

Biodiesel cetane number: Based on the above reference there exists a relation between cetane number and properties of biodiesel so a correlation was developed which is given as equation:

CN=K5+K4 v+K3 HV+K2 FP+K1p (5)

Where K1, K2, K3, K4, K5 ARE CONSTANTS and v is kinematic viscosity (mm2/sec), HV is heating value (MJ/Kg), FP is flash point (ºC) p is density.

Determination of the % conversion of biodiesel: The conversion of olive waste oil to

biodiesel using the following Equation:

Yield (%) =weight of the biodiesel (g) /weight of the olive waste oil (g) x100 (6)

Data analysis: At least three replications for each oil sample were performed with each test. The averages and standard deviation were calculated by statistical analysis using SPSS program 10.0 (IBM Corporation, Armonk, Ny). The differences were considered level significant when P < 0.05 at a confident level of 95%. Arrangement of data for statistical analysis was performed by using Microsoft Office Excel (2007).

3. RESULTS AND DISCUSION

Olive waste oil analysis: The oil content in the olive waste was 15.20. The acid value as % of oleic acid was 44.00 mg KOH/g oil. The peroxide value of the oil was measured as 189.00 meq.O_2 /Kg oil these values were higher than those required by Regulation EC/1989/2003 (Moya *et al.,* 2006) (Acidity < 0.8%; peroxide value < 20 meq.O_2/kg oil). Thus, oil is said to be virgin lampante olive oil and do not fit for human consumption. For high free fatty acid content oil, an acidic catalyst can be used initially to convert free fatty acid level to the esters and to decrease the free fatty acid level 0.5%, and then the transesterification of oil can performed using an alkaline catalyst.

Biodiesel production: *1-First step* (free fatty acid esterification: H_2SO_4 was used to esterify and reduce free fatty acid content of olive waste oil from 44.00 To less than 1.00% (acid value mg KOH g oil. The pretreatment step was carried with different catalyst concentrations: 2% and 10% of oil, and methanol rations (20 and 40% v/v). All tests were performed at constant reaction temperature of 60ºC. The results indicated that with catalyst concentrations 2%v/v and methanol to oil ratio of 20%, it was possible to reduce the free fatty acid content from 44.00 % (mg KOH/g oil) to 1.5%. The free fatty acid content of the treated oil was lower when the methanol to oil ratio increased to 40% v/v. increasing the acid concentration reduced the free fatty acid content of the oil. In order to reduce the free fatty acid to free fatty acid level to 0.5%, an H_2SO_4 concentration of 10 %v/v of oil was required. The effect of the catalyst on free fatty acid conversion is shown in Table 1. The H_2SO_4 can catalyze methanolysis of free fatty acid but cannot catalyze the transesterification of triglycerides. So the acid value can be calculated for the conversion of free fatty acid. In this Table it can be seen the effect of the catalysts on the free fatty acid esterification with methanol at the optimal reaction conditions

determined in this investigation. The conversion of free fatty acid was 98.86% when H_2SO_4 was used as catalyst. The acid value of the olive waste oil decreased the presence of H_2SO_4.

Table 1 Effect of the catalysts on the olive waste oil acid value after the free fatty acid esterification

Run	Initial acid value (%)	H_2SO_4 (v %)	Final acid value (%)	Free acid a conversion (%)
1	44.00	0	44.00±2.1	0.00±0.00
2	44.00	2	1.51±0.01	96.55±4.15
3	44.00	5	0.78±0.11	98.22±5.00
4	44.00	10	0.50±0.07	98.82±5.19

The results (mean ± standard deviation from triplicate runs) (P value ≥0.05)

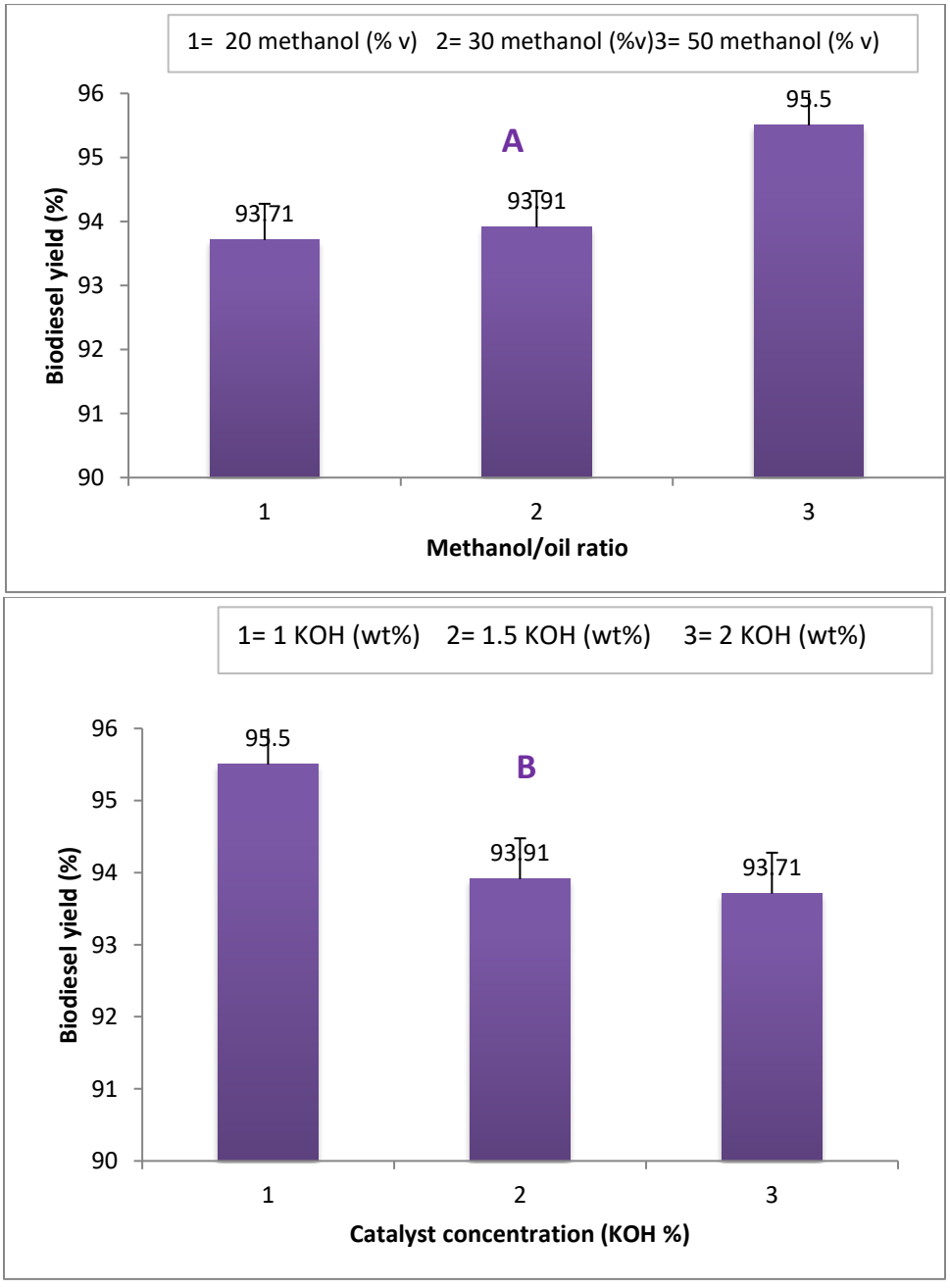

Figure 1 Influence of the methanol/oil ratio (A) and effect of catalyst concentration (B) on biodiesel production. The results (mean ± standard deviation from triplicate runs) (P value ≥0.05)

2- Second step: alkali catalyzed transesterification of olive waste oil: The biodiesel obtained from the potassium hydroxide catalyzed transesterification of olive waste oil with methanol after the free fatty acid esterification catalyzed by H_2SO_4 with methanol was characterized by its physical and chemical properties (Table 2).

In order to investigate the different operating parameters on the conversion of olive waste oil to biodiesel, the reaction was carried out with different methanol-to-oil ratios (20, 30, 50 % v/v), different catalyst concentration (1, 1.5, 2 wt. %) and reaction temperature (60ºC). The reaction time in all experiments was set constant (4 hr.).The experimental conditions were presented in Table 2 together with the calculated conversion of olive waste oil to biodiesel (methyl ester) using the following equation (6).

The methanol/oil ratio is one of the most important factors affecting the yield of biodiesel. The transesterification is usually carried out with an extra amount of methanol in order to shift the equilibrium to the direction of methyl ester formation. The methanol/oil ratio was varied (20, 30, 50%v/v).The maximum methyl ester conversions for olive waste oil was 95.50% at a ratio of methanol/oil at 50% (Figure 1, A).

Three levels of catalyst concentration were selected; the reactions were conducted at 1.00, 1.50, 2.00wt%. Selection of the levels was carried out based on results obtained by other investigators who optimized biodiesel production via alkali-catalyzed transesterification from various oils using these concentrations (Rashid *et al.,* 2008; Leung and Guo 2006 and Vicente *et al.,* 1998). The production of biodiesel was found to be highly dependent on the catalyst concentration. Transesterification of olive waste oil with 1.00% gave the best conversions (Figure 1, B). When the concentration of catalyst exceeded 1.50%, the biodiesel production decreased due to excess of catalyst can also cause hydrolysis; saponification and formation of emulsions block the reaction (Lin *et al.,* 2009).

Table 2 Influence of operating variables on the oil to biodiesel (methyl ester) conversion in alkali-catalyzed transesterification of olive waste oil

Run	Treated olive waste oil(g)	Methanol (V %)	KOH (wt. %)	Temperature	Biodiesel (g)	Yield (%)
1	49.30±4.81	20	2.00	60	46.20±4.01	93.50±6.66
2	56.33±5.54	30	1.50	60	52.90±5.32	93.91±6.32
3	60.30±6.21	50	1.00	60	57.59±4.99	95.50±7.55

The results (mean ± standard deviation from triplicate runs) (P value ≥0.05)

The results are shown in Table 3. The fatty acid methyl ester in the biodiesel obtained was analyzed using gas chromatography. The quantitative and qualitative analysis of the compounds contained in the biodiesel is listed in Table 3. After alkali-catalyzed transesterification the biodiesel showed the following fatty acid methyl esters composition: methyl palmitate (16:0), methyl stearate (18:0), methyl oleate (18:1), methyl linoleate (18:2) and methyl linolenate (18:3), with small amounts of other methyl esters also present. Olive waste oil biodiesel is rich in methyl oleate and the presence of monounsaturated methyl esters gives it a high cetane number which is one of its main advantages compared to conventional diesel fuels The transesterification reaction of the triglycerides in the olive waste oil after the esterification process was performed with potassium hydroxide homogeneous catalyst. Table 4 reports the various values determined from the analysis the fatty methyl esters produced. These values show that the process proposed in this investigation can generate biodiesel of a good quality. Olive waste oil biodiesel has a cetane number of 56.00 exceeding those of other oils such as sunflower oil (49.00), soybean oil (47.00) and cottonseed oil (51.00) (Leung *et al.,* 2010), what it would provide higher combustion efficiency. The viscosity is also one of the most important properties of biodiesels science it affects the operation of fuel injection equipment. Low viscosity leads to better atomization of the fuel spray and more accurate operation of the fuel injectors (Demirbas, 2009).The kinematic viscosity of the olive waste oil biodiesel is 5.00 mm2/s at 40ºC. This process prevents the water waste streams, excess of methanol, high pressure and high cost stainless steel equipment used to eliminate the homogeneous acid catalyst that would be used for the free fatty acid esterification step in the olive waste oil. The percentage conversion of triglycerides to methyl esters using Equation 6 was found to 95.50%.

Table 3 Biodiesel composition determined by gas chromatography

Compound	Content (%)
Methyl butyrate	0.04±0.00
Methyl hexanoate	0.11±0.00
Methyl ocatnoate	0.08±0.00
Methyl tridecanoate	0.08±0.00
Methyl pentadecanoate	0.02±0.00
Methyl palmitate	16.20±1.11
Methyl stearate	4.02±0.56
Methyl oleate	70.08±7.89
Methyl linoleate	12.14±0.88
Methyl linolenate	0.44±0.23

The results (mean ± standard deviation from triplicate runs) (P value ≥0.05)

Table 4 Physical and chemical properties of fatty acid methyl ester obtained from olive waste oil

Property	Levels
Density at 25ºC	870.00±20.67
Kinematic viscosity at 40ºC	5.00±0.39
Acid number	0.30±0.09
Cetane number	56.00±3.96
Heating value (MJ/Kg)	44.50±2.76
Flash point ºC	57.00±4.98

The results (mean ± standard deviation from triplicate runs) (P value ≥0.05)

4. CONCLUSION

Biodiesel production costs are rather high compared to petroleum based diesel fuel. The use of olive waste oil as feedstock can lower the cost for biodiesel production. This investigation showed that the use of H_2SO_4 as esterification catalyst resulted in a strong decrease of the free fatty acids present in the olive waste oil used as feedstock for the biodiesel production. The second step transesterification reaction present of triglycerides in the olive waste oil catalyzed by KOH produced biodiesel a high content of methyl esters.

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